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# Solution-polymerized SBR — Evaluation methods of viscoelastic properties

*SBR polymérisé en solution — Méthodes d'évaluation des propriétés viscoélastiques*



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# Foreword

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This document was prepared by Technical Committee ISO/TC 45, *Rubber and rubber products*, Subcommittee SC 3, *Raw materials (including latex) for use in the rubber industry*.



Any feedback or questions on this document should be directed to the user's national standards body. A complete listing of these bodies can be found at www.iso.org/members.html.

# Introduction

A variety of solution-polymerized styrene-butadiene rubber (S-SBR) have been developed and used for tires because they show excellent balance between rolling resistance and wet grip.

It is well known that functionalized S-SBR can strongly react with fillers, resulting in better dispersion and contributing to the low rolling resistance tire. These phenomena can be explained by the viscoelastic properties.

In other words, in order to describe the performance of S-SBR, it is necessary to evaluate viscoelastic properties. Therefore, a standard specifying the model compound formulation, mixing procedure, sample preparation, test conditions, etc. for evaluating the viscoelastic properties of the S-SBR compound is useful to the rubber industries.

# Solution-polymerized SBR — Evaluation methods of viscoelastic properties

**WARNING — Persons using this document should be familiar with normal laboratory practice. This document does not purport to address all of the safety problems, if any, associated with its use. It is the responsibility of the user to establish appropriate safety and health practices and to determine the applicability of any other restrictions.**

## 1 Scope

This document specifies the standard test formulation, mixing procedure and test methods for evaluation of viscoelastic properties in a compound based on solution-polymerized styrene-butadiene rubber (S-SBR), including functionalized S-SBR[7].

## 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.



ISO 289-1, *Rubber, unvulcanized — Determinations using a shearing-disc viscometer — Part 1: Determination of Mooney viscosity*

ISO 2393, *Rubber test mixes — Preparation, mixing and vulcanization — Equipment and procedures*



ISO 4664-1, *Rubber, vulcanized or thermoplastic — Determination of dynamic properties — Part 1: General guidance*

ISO 6502-2, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 2: Oscillating disc curemeter*

ISO 6502-3, *Rubber — Measurement of vulcanization characteristics using curemeters — Part 3: Rotorless curemeter*

ISO 23529, *Rubber — General procedures for preparing and conditioning test pieces for physical test methods*

## 3 Terms and definitions

For the purposes of this document, the terms and definitions given in ISO 4664-1 apply.

ISO and IEC maintain terminology databases for use in standardization at the following addresses:

— ISO Online browsing platform: available at https://www.iso.org/obp

— IEC Electropedia: available at https://www.electropedia.org/

## 4 Test formulation

The standard test formulation given in Table 1 shall be used for the evaluation of any type of S-SBR.

Formulation A is an oil-extended S-SBR formulation. Formulation B is a non oil-extended S-SBR formulation.

International or national standard chemicals shall be used if available. Materials used shall be chemically equivalent to those indicated in Table 1.

**Table 1 — Standard test formulation**

| Material | pphr (parts per hundred rubber)[a] | |
|---|---|---|
| | Formulation A | Formulation B |
| **First stage** | | |
| S-SBR, extended with oil | 70 + $X$[b] | — |
| S-SBR | — | 70 |
| BR[c] | 30 | 30 |
| Silica, precipitated[d] | 70 | 70 |
| Carbon black N234 | 10 | 10 |
| Silane coupling agent TESPD[e] | 5,6 | 5,6 |
| ZnO[f] | 3 | 3 |
| Stearic acid[g] | 2 | 2 |
| Oil TDAE[h] | 31,25 - $X$ | 31,25 |
| Antioxidant (6PPD)[i] | 1,5 | 1,5 |
| Wax[j] | 1,0 | 1,0 |
| **Second stage** (no additional ingredient) | | |
| **Third stage** | | |
| DPG[k] | 2 | 2 |
| CBS[l] | 1,5 | 1,5 |
| Sulfur[m] | 2,0 | 2,0 |
| Total | 229,85 | 229,85 |

[a] The abbreviation, pphr, is defined in ISO 1382:2020, 3.345.

[b] $X$ = parts of oil, mass fraction, per 100 parts of base polymer in the oil-extended rubber.

[c] Butadiene rubber, manufactured with neodymium or cobalt catalyst, 96 % (mass fraction) *cis* 1,4 content.

[d] The CTAB surface area of 150 $m^2/g$ to 180$m^2/g$ is used. The CTAB is measured according to ISO 5794-1.

[e] Bis(triethoxysilylpropyl)disulfide.

[f] Zinc oxide, indirect type, class B1a according to ISO 9298:2017, Table D.1.

[g] Stearic acid, stearic/palmitic 65/30, class B according to ISO 8312:2015, Table L.1.

[h] Treated distillate aromatic extract.

[i] *N*-(1,3-Dimethylbutyl)-*N'*-phenyl-*p*-phenylenediamine.

[j] Wax, mixture of refined hydrocarbons.

[k] *N,N'*-Diphenylguanidine.

[l] *N*-Cyclohexyl-2-benzothiazylsulfenamide.

[m] Sulfur, soluble (rhombic), grade W according to ISO 8332:2018, Table A.1.



## 5 Procedure for sample preparation

### 5.1 General

Equipment and procedure for preparation, mixing and vulcanization shall be in accordance with ISO 2393. The following procedure is an example which has been found suitable for 600 $cm^3$ laboratory internal mixer.

At the beginning of each series of test mixes, a machine conditioning batch shall be mixed using the same formulation as the mixes under test.

## 5.2 First stage

— Filling factor: 0,70.

— Rotational speed: 50 r/min.

— Starting temperature: 90 °C ± 3 °C.

— Friction: 1:1,14.

— Ram pressure: 0,5 MPa.

The first stage procedure is given in 5.2 a) to 5.2 f).

The temperature of the batch discharged on completion of mixing shall be between 155 °C and 165 °C. If necessary, adjust starting temperature, rotation speed and/or filling factor to reach discharge temperature.

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Adjust the temperature of the laboratory internal mixer to a starting temperature of 90 °C ± 3 °C. Close the discharge door, set the rotor speed and raise the ram. | — | — |
| b) | Load the rubber, lower the ram and allow the rubber to be masticated. | 0,5 | 0,5 |
| c) | Raise the ram and load the half of the silica, silane coupling agent, oil. Lower the ram and allow the batch to mix. | 1,5 | 2,0 |
| d) | Raise the ram and load the half of the silica, antioxidant, wax, ZnO, stearic acid, carbon black. Lower the ram and allow the batch to mix. | 1,5 | 3,5 |
| e) | Raise the ram and clean the mixer throat and the top of the ram. Lower the ram and allow the batch to mix. | 1,5 | 5,0 |
| f) | Discharge the batch (batch temperature: 155 °C to 165 °C). | | |



After discharging the batch, immediately check the temperature of the batch with a suitable temperature measuring device. If the temperature as measured falls outside the range of 155 °C to 165 °C, discard the batch. Pass the batch three times through a mill with a mill opening of 2,5 mm and a roll temperature of 50 °C ± 5 °C.

Determine the mass of the batch. If the mass differs from the theoretical value by more than +0,5 % or –1,5 %, discard the batch and remix.

Leave the batch for at least 3 h and up to 24 h at room temperature, if possible, at standard temperature and humidity as defined in ISO 23529.

## 5.3 Second stage

— Filling factor: 0,69.

— Rotational speed: 50 r/min.

— Starting temperature: 90 °C (adjust so that the discharge temperature is 155 °C to 165 °C).

— Friction: 1:1,14.

— Ram pressure: 0,5 MPa.

The second stage procedure is given in 5.3 a) to 5.3 c).

| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Plasticize the batch from the first stage. | 2,0 | 2,0 |
| b) | Maintain the batch temperature of 160 °C by adjusting the starting temperature. | 3,0 | 5,0 |
| c) | Discharge the batch (batch temperature: 155 °C to 165 °C). | | |

Pass the batch three times through a mill with a mill opening of 2,5 mm and a roll temperature of 50 °C ± 5 °C.

Leave the batch for at least 3 h and up to 24 h at room temperature before proceeding to the third stage, if possible, at standard temperature and humidity as defined in ISO 23529.

### 5.4 Third stage

— Filling factor: 0,70.

— Rotational speed: 30 r/min.

— Starting temperature: 50 °C ± 3 °C.

— Friction: 1:1,14.

— Ram pressure: 0,5 MPa.



The third stage procedure is given in 5.4 a) to 5.4 e).



| | | Duration (min) | Cumulative time (min) |
|---|---|---|---|
| a) | Adjust the temperature of the laboratory internal mixer to a starting temperature of 50 °C ± 3 °C. Close the discharge door, set the rotor speed and raise the ram. | — | — |
| b) | Load the batch of the second stage, lower the ram and allow the rubber to be masticated. | 0,5 | 0,5 |
| c) | Raise the ram and load DPG, CBS, and sulfur. Lower the ram and allow the batch to mix. | 1,0 | 1,5 |
| d) | Raise the ram and clean the mixer throat and the top of the ram. Lower the ram and allow the batch to mix. | 0,5 | 2,0 |
| e) | Discharge the batch (batch temperature: 90 °C to 110 °C). | | |

The batch is transferred to mill. Form a sheet for 20 s with a mill opening of 3 mm to 4 mm between the rolls. During the next 40 s, make 3/4 cuts three times from each side. Cut out the batch from the mill. Set the mill opening at 0,8 mm and pass the rolled batch endwise through the rolls six times, introducing it from each end alternately. Finally, form one sheet of the thickness 2,2 mm.

Condition the batch at least 24 h at room temperature after mixing and prior to vulcanizing, if possible at standard temperature and humidity as defined in ISO 23529.

## 6 Testing of the uncured mix

Determine the viscosity at 100 °C using the shearing disc viscometer in accordance with ISO 289-1.

# 7 Evaluation of vulcanization characteristics

## 7.1 General

Evaluation of vulcanization characteristics is carried out according to either 7.2 or 7.3.

## 7.2 Evaluation according to oscillating disc curemeter test

Measure the following standard test parameters:

$M_L$, $M_H$, $t_c'$ (10), $t_c'$ (50) and $t_c'$ (90)

in accordance with ISO 6502-2, using the following test conditions:

— oscillation frequency: 1,7 Hz (100 cycles per minute);

— amplitude of oscillation: ±1° arc;

— selectivity: to be chosen to give at least 75 % full scale deflection at $M_H$;

— die temperature: 160,0 °C ± 0,3 °C;

— pre-heat time: none.

## 7.3 Evaluation according to rotorless curemeter test



Measure the following standard test parameters:

$M_L$, $M_H$, $t_c'$ (10), $t_c'$ (50) and $t_c'$ (90)

in accordance with ISO 6502-3, using the following test conditions:



— oscillation frequency: 1,7 Hz (100 cycles per minute);

— amplitude of oscillation: 0,5° arc;

— selectivity: to be chosen to give at least 75 % full scale deflection at $M_H$;

— die temperature: 160,0 °C ± 0,3 °C;

— pre-heat time: none.

# 8 Evaluation of dynamic viscoelastic properties

## 8.1 General

Regarding the dynamic viscoelastic properties, two kinds of evaluations, the strain dependency (see 8.4) and the temperature dependency (see 8.5), shall be carried out.

In the strain dependency, it is possible to evaluate the change in the filler network structure, that is, the dispersibility of the filler in a compound. In temperature dependency, temperature dependency of complex modulus, including glass transition temperature and mobility of molecular chain can be evaluated.

The strain dependency of the filling compound is known as the Payne effect[8][9] (see Annex D) and reflects the filler networks. The difference in absolute value of complex modulus between low strain and high strain is conventionally used as an indicator of the filler dispersibility. Especially, it is known that when the polymer type is changed to the functionalized polymer, the difference value greatly decreases. It is useful as an indicator of filler dispersibility by polymer types. See Figure C.1.